Evaluation of the Viscoelastoplastic Properties of a Clay Material Using a Flow Curve

S. Paumier[1,2*], A. Pantet [1], P. Monnet [1] and N. Touze-Foltz [2]

[1] Laboratoire d'Etudes Aérodynamiques (LEA), ENSMA, CNRS, Téléport 2, Boulevard Curie, 86962 Chasseneuil, France
[2] Hydrosystems and Bioprocesses Research Unit, Cemagref, B.P. 44, 92163 Antony Cedex, France

* Email: sandrine.paumier@cemagref.fr
Fax: x33.1.40966270

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Abstract:
The specific properties of bentonite such as hydration, swelling, water absorption, viscosity, yield stress and thixotropy make it a valuable material in the form of mineral powder for a wide range of uses in agronomy, cosmetics and civil engineering. A flow curve is a quick test used to evaluate the rheological basic properties of a viscous fluid. However, many bentonite dispersions exhibit a complex flow curve, with yield stress and thixotropy area, especially at high concentration. In this study, flow curves from raw and activated bentonites dispersed in water were acquired at 6, 8 and 10% mass concentrations. Five stages along the flow curve were identified. To explain each stage, rheograms obtained from a dispersion made with a model material were studied in depth. The model material was a smectite extracted from a raw bentonite then saturated with calcium or sodium. Many homoionic and bi-ionic dispersions were prepared at various concentrations. The analysis and the modelling of some creep-recovery tests by a Zener model showed the relationship between the initial stage in the flow curve, named AB stage, and the viscoelastic properties of the fluid. The AB stage corresponds to the deformation of the material in the solid state. τ_B point corresponds to an intermediate yield stress between the solid state and the start of the heterogeneous fracturation. The study of many bi-ionic dispersions allowed drawing the evolution of the yield stress as a function of concentration and saturation. The composition of the raw bentonites was expressed as an equivalent bi-ionic dispersion by calculating an active smectite percentage. A good correlation was obtained at the highest concentrations between τ_B from the bi-ionic model dispersions and the raw bentonites dispersions.

Zusammenfassung:
Seine spezifischen Eigenschaften (z. B. Hydratation, Schwellverhalten, Wasserabsorption, Viskosität, Fließspannung und Thixotropie) machen Bentonit zu einem wertvollen Material in der Form eines mineralischen Pulvers für viele Anwendungen im Bereich der Agrarwissenschaft, Kosmetik und des Bauingenieurwesens. Eine Fließkurve ist ein schneller Test, um die rheologischen Eigenschaften als viskoses Fluid zu testen. Jedoch weisen viele Bentonit-Dispersionen ein komplexes Fließverhalten mit Fließgrenze und thixotropem Verhalten auf, insbesondere bei hohen Konzentrationen. In dieser Arbeit wurden die Fließkurven unbehandelter und aktivierter lang der Fließkurven wurden identifiziert. Um jedes Stadium zu erklären, wurden Rheogramme einer Dispersion eines Modell-Materials untersucht. Die Modellsubstanz war ein Smektit, der aus unbehandeltem Bentonit extrahiert wurde und dann mit Kalzium bzw. Natrium gesättigt wurde. Viele homoionische und bi-ionische Dispersionen mit unterschiedlichen Konzentrationen wurden hergestellt. Die Analyse und die Modellierung einiger Kriech-Erholungskurven mit Hilfe des Zener-Modells verdeutlichen die Beziehung zwischen dem Anfangsstadium der Fließkurven (AB-Stadium) und den viskoelastischen Eigenschaften des Fluids. Das AB-Stadium entspricht der Deformation des Materials im festen Zustand. Der Punkt B entspricht einer intermediären Fließspannung zwischen dem festen Zustand und dem Beginn der heterogenen Brüche. Die Untersuchung vieler bi-ionischer Dispersionen erlaubt die Darstellung der Fließspannung als Funktion der Konzentration und der Sättigung. Die Zusammensetzung unbehandelter Bentonite wurde durch eine äquivalente bi-ionische Dispersion mit Hilfe der Berechnung eines aktiven smektischen Gehalts beschrieben. Eine gute Korrelation wurde bei den höchsten Konzentrationen zwischen dem Punkt B der bi-ionischen Modelldispersion und den unbehandelten Bentonit-Dispersionen erhalten.

Résumé:
Les bentonites sont des matériaux naturels contenant de la smectite. Cette argile est susceptible d'adsorber de l'eau pour créer des gels aux propriétés rhéologiques couramment utilisées en agronomie, en cosmétique ou dans le domaine du génie civil. La réalisation de courbes d'écoulement permet d'évaluer les propriétés rhéologiques de base d'un fluide visqueux. Toutefois de nombreuses suspensions de bentonites révèlent des courbes

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for each bentonite using Eq. 10. Values are presented in Table 4.

$$ASP(\%) = \frac{SWP.RSR}{100} \quad (10)$$

where SWP (%) is the smectite weight percentage and RSR (%) is the removable sodium ratio. SWP and RSR values are given in Table 2.

The ASP is an approximate value that allows comparing the raw bentonites with a composition of bi-ionic smectite, expressed with the ESP. One can notice that standard NF X31-108 used to determine the RSR did not include any washing stage. Furthermore the RSR probably contained exchangeable cations and a part of soluble salts. However, the process was brief (only one hour) and the dissolution of the soluble salts may be low. The natural sodium bentonites LX2 and LX3 could be compared with bi-ionic smectite dispersions at 45ESP and 48ESP, respectively. The sodium-activated bentonites LX5 and LX8 could be compared with bi-ionic dispersions at 67ESP and 62ESP, respectively (Tab. 4). The yield stress for the natural sodium bentonite dispersions are very closed to those found for the bi-ionic smectite dispersions. Moreover, the yield stresses of the sodium activated bentonites dispersions are higher than those of the bi-ionic dispersions. At 10%, the results are in the same order of magnitude. Other parameters, such as soluble salts introduced during the activation or mineralogical parameters probably have a larger influence on the yield stresses than ASP.

5 CONCLUSIONS

Rheometry can be used to classify the flow properties and the solid viscoelastic properties of bentonites dispersions using only flow curves. The interesting point is that the flow curves are classical tests, easily executable with a rheometer of middle class quality; they do not require an expensive material to perform dynamic tests. The A-B stage clearly corresponds to the solid state. The A-B stage is well reproduced and can be exploited even if the flow at $\tau > \tau_B$ is heterogeneous. The A-B stage can provide information on the viscoelastic properties of the dispersion in solid state without a large trend of tests. The shear stress at point B corresponded to the yield stress evaluated with some classical creep-recovery curves.

The application of this method for determination of the yield stress value for raw bentonites and a model smectite allowed establishing the following results: the extracted and saturated smectites could be compared with the raw bentonites using the exchangeable sodium percentage and active sodium percentage, respectively. The results for the sodium natural bentonites are comparable to the model smectite. The results for the sodium-activated bentonites are more complex and chemical or mineralogical parameters may influence the yield stress value. The activation process gave rise to higher yield stresses than the equivalent bi-ionic smectite.

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